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INVESTIGATION OF INTEGRALLY COMPOSED VARIABLE ENERGY GAP PHOTOVOLTAIC SOLAR ENERGY CONVERTER

THE EAGLE-PICHER RESEARCH LABORATORIES

Chemicals & Metals Division

Miami, Oklahoma

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FINAL TECHNICAL SUMMARY REPORT

Covering the Period -

February 9, 1961 - February 9, 1962

Report Date: February 10, 1962

"INVESTIGATION OF INTEGRALLY COMPOSED VARIABLE ENERGY GAP PHOTOVOLTAIC SOLAR ENERGY CONVERTER"

ARPA Order Number 80-61

Date of Contract: February 9, 1961

Amount of Contract: $34,592.00

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The work performed under this Contract was made possible by the support of the Advanced Research Projects Agency Order Nr. 80-61 through the U. S. Army Signal Research & Development Laboratory.

I. PURPOSE

The general objective of this work was to fabricate and study an integrally composed variable energy gap solar cell, and to determine the feasibility of improving the photovoltaic properties of solar cells through the use of the variable gap principle. Specific objectives were to (1) increase the conversion efficiency, (2) improve the temperature performance, and (3) demonstrate the high energy gap "window" effect which may improve collection efficiency and lower the sheet resistance. The variable gap solar cell under study is fabricated of two III-V compounds, gallium phosphide and gallium arsenide, in which the top surface is composed largely of gallium phosphide, forming a "window" of higher band gap than the gallium arsenide portion of the cell. The practical objective of the work was to demonstrate any advantageous characteristics of such a cell, such as better temperature performance, efficiency, or radiation resistance, for special applications when the state-of-the-art, material-wise, makes gallium arsenide competitive with present materials, and to demonstrate the feasibility of using such a "window" of higher band gap to achieve higher collection efficiency and lower sheet resistance.

II. ABSTRACT

Gallium phosphide as a discrete phase was produced by solid state diffusion of phosphorus into gallium arsenide. High concentrations of gallium phosphide, of the order of 90 percent, have been determined by x-ray analysis. Layers of such gallium phosphide have been defined by etch staining and photographed. Thickness of 50 microns have been obtained.

Some knowledge of the conversion process has been obtained, allowing postulation of a step-type conversion mechanism, which is discussed in this report.

Tentative data indicates a non-linear diffusion-conversion rate, with rapid rates initially and very slow end rates for a given time of diffusion.

The diffusion rate of zinc in gallium phosphide layers was observed to be significantly higher than in gallium arsenide. At least an order of magnitude, perhaps several orders of magnitude, was noted.

Two general categories were defined as bracketing the various possible depths, concentrations and gradients of variable gap cells. They represent (a) very thin, low concentration gallium phosphide surfaces, 1 to 2 microns thick, and (b) thick, high concentration gallium phosphide surfaces 2 to 10 microns thick. They were observed to exhibit dissimilar characteristics, especially in spectral response.

Good efficiencies and short circuit currents per unit area have been obtained in the thin layer cells. Indications of improved high temperature performance, in this type cell, was observed.

Spectral response data supports the principle of the high band gap "window" effect in the thick species of cell.

An electro-etch-stain technique produces excellent definition of p-n junctions and gallium phosphide layers without destruction of the cell.

Type conversion of bulk gallium arsenide material from n-type to p-type was observed during the phosphorus diffusion for periods of 340 hours at 900°C.

III. PUBLICATIONS, CONFERENCES, & REPORTS

Conferences.

A Conference was held at Eagle-Picher Research Laboratories, on February 14, 15, and 16, 1961, with Mr. L. Schwartz of Evans Laboratories, Fort Monmouth, New Jersey, and Messrs. W. E. Medcalf and Louis E. Stone of this laboratory. The subject of the conference was the direction and emphasis of the projected investigation. Methods of fabrication, testing, and evaluation were discussed and an outline of future work agreed upon. The conference was useful and informative.

A second conference was held on March 30,1961 at Evans Laboratories, Fort Monmouth, New Jersey. Present were Messrs. Wm. Cherry, J. Mandelkorn, L. Schwartz, J. Kesperis, and Louis E. Stone. The subject of the conference included current progress of studies, formulation of testing procedures with regard to elevated temperature tests, integrated red response of the variable gap cell, and diode testing techniques. At this conference, ten sample specimens of gallium arsenide wafers diffused in varying degree with phosphorus were delivered. The conference was useful and lent direction to further efforts.

The Annual Power Sources Conference was held at Atlantic City, New Jersey, on May 9, 10, and 11. It was attended by Mr. Stone of this laboratory.

Following the Power Source Conference, a private conference was scheduled and held at Evans Laboratories on May 12. At this conference, Mr. Mandelkorn mentioned that a discrete phase of gallium phosphide had been

identified on at least one of the sample specimens delivered March 29th.- corroborating our own x-ray determination of a previous date. Further specimens (4) of cells were delivered at this time.

A conference was held at Eagle-Picher Research Laboratories, Miami, Oklahoma on July 25 and 26th., with Mr. Laurence Schwartz, of Evans Laboratories, and Messrs. W. E. Madcalf and Louis E. Stone, of this laboratory, The subject of the conference was the direction and emphasis of projected work, and the current progress of the investigation. Current state of progress specimen cells were delivered to Mr. Schwartz at this meeting.

A conference was held on December 5, 1961, at Evans Laboratories, with Messrs. Wm. Cherry, L. Schwartz, and J. Mandelkorn, of Evans Laboratories, and Messrs. W. E. Medcalf and Louis E. Stone of Eagle-Picher Company. The purpose of the conference was again the current progress and direction of the investigations. Another group of specimen cells was delivered, and visual microscopic examination of other specimens were demonstrated.

A conference was held on January 24, 1962, at Evans Laboratories with Messrs. L. Schwartz and J. Mandelkorn. Messrs. K. E. Bean and Louis E. Stone were present from The Eagle-Picher Company. Spectral response data, correlation of light measurements, junction and surface characteristics were discussed and guide lines established for the remainder of the contract period.

Reports.

Monthly Letter-Type reports have been submitted on or before the 4th. day of each month, as per contractual agreement.

The First Semi-Annual Technical Summary Report was submitted on July 10, 1961.

The Second Semi-Annual Technical Summary Report was submitted January 10, 1962.

Publications.

A brief article, entitled "Conversion of Gallium Arsenide to Gallium Phosphide by Solid State Diffusion" has been submitted as a Letter to the Editor, Journal of Applied Physics, for publication.

IV. FACTUAL DATA

A. Introduction.

The theoretical basis for the research performed under this contract has been discussed in detail in previous reports[1]. Briefly the gallium arsenide-gallium phosphide cell would have a surface composed primarily of gallium phosphide, diminishing in concentration with increasing depth. The higher energy gap layer allows transmission of lower energy photons to a depth nearer the junction, improving collection and hence efficiency. The greater depth, when diffused with zinc as a p-type dopant, also reduces sheet resistance. The higher band gap material shows improved high temperature characteristics from a theoretical viewpoint. Emphasis was placed on the demonstration of such effects, including the "window effect" of the gallium phosphide layer. Inherent in the stated purpose was the study of the diffusion-conversion process to accomplish the variable gap structure, and derive controls for such processes and techniques which were required.

B. Approach to the Problem:

The approach to the problem included the following:

1. A study of gallium arsenide crystals to obtain and improve starting materials.
2. A study of the diffusion-conversion process to accomplish the desired variable gap structure. This necessarily encompassed knowledge and control of the conversion mechanism to obtain discrete phase gallium phosphide rather than interstitially diffused phosphorus. It also implies determination of the optimum thickness and concentration of the gallium phosphide layer.

3. A study of the fabrication of variable gap cells, of the desired structure, and of single gap cells as a reference for comparison. This encompassed the following:

   (a). Techniques of forming p-n junctions in both types of cells, with emphasis in control of depth, density of dopant, and homogeneity of the diffused layer.

   (b). Identification and measurement of the gallium phosphide layer.

   (c). Identification and measurement of the depth of the p-n junction.

   (d). Electroding and contacting both type cells to obtain ohmic, low resistance contacts stable to temperatures of about 200°C.

4. Evaluation of Cells.

The evaluation of cells was of prime importance. Routine analysis included the following:

(a). V_{oc}, I_{sc}, and conversion efficiency.
(b). Spectral response.
(c). Diode characteristics.
(d). Temperature coefficients of V_{oc}, I_{sc}, Eff_{mp}.

Other parameters considered to be significant included:

(a). Carrier concentration.
(b). Sheet resistance.
(c). Contact resistance.

5. Evaluation of Starting Material.

It was considered important to establish the effect of the following material characteristics on cell performance:

(a). Resistivity.
(b). Mobility.
(c). Minority carrier lifetime.
(d). Crystal orientation.
(e). Gallium or arsenic face of the crystal.

C. Phosphorus Diffusion.

A major effort was made during this contract period to determine and clarify the mechanism involved in the conversion of gallium arsenide to gallium phosphide by solid state diffusion. This effort was required because the effect of varying phosphorus diffusion schedules was observed to change the photovoltaic parameters significantly.

The DeBye-Scherrer powder diffraction technique has been widely used for the desired determination. Disadvantages of mechanical removal of such a thin layer from the surface of a cell was overcome by including prepared known size powder specimens in the ampoule during diffusion as previously reported[2]. Full data on a series of such determinations follow in Table I.

TABLE I

Composition of Phosphorus Diffused Gallium Arsenide Powder, based on X-Ray Studies.

Sample	M6108 BE	M6108 BF	M6108 BG	M6108 BH
Time,	2 Hours	20 Hours	50 Hours	50 Hours
Temperature °C,	800°C	800°C	800°C	900°C
Pressure,	15 Atm.	15 Atm.	15 Atm.	20 Atm.
Percent Gallium Phosphide Content of-				
Arsenic-Rich Phase,	4.2%	38.6%	N. D.	1.5%
Phosphorus-Rich Phase,	98.0%	99.5%	86.1%	93.1%
Strongest Intermediate Solid Solution Phase,	49.8%	86.1%	N. D.	N. D.
Gallium Phosphide Composition Calculated of all phases,	50.8%	81.4%	85.0%	88.5%

NOTE: "N.D." = Not Detected.

Figure 1 illustrates the appearance and color change in the above series of powder specimens. There was good correlation between the color changes and the gallium phosphide content as determined by x-ray.

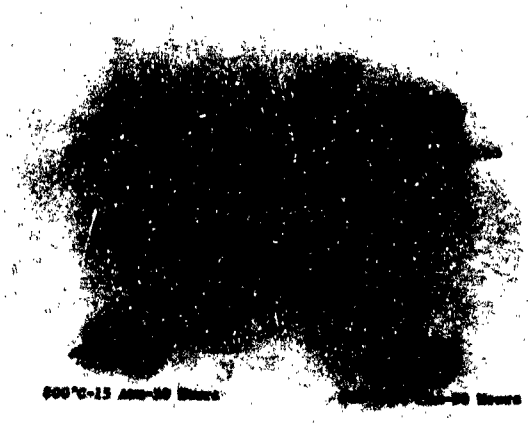


Figure 1. Color Change of Phosphorus Diffused Gallium Arsenide Powder as a Function of Diffusion Schedules.

Some discussion of the x-ray data is in order. The pattern of the more heavily diffused specimens was normal, well defined, and sharp. The pattern of the more lightly diffused specimen, M6108 BE, however, was that of two major phases of similar crystal structure with edge lengths differing by only about 0.1 A°. This resulted in a repeated pattern of lines, as would be expected. However, the space between the lines of the two patterns are filled with sharply defined lines, or striae, which are very distinct on the larger spacings (low Theta angles). These intermediate lines come from well defined crystalline phases, evidently of

compositions intermediate between the two major phases that are the end members of the solid solution system present in the sample.

The line structures were also of interest. The line of the pattern from the arsenic-rich end member is very grainy, indicating a particle size at least an order, perhaps 2 or 3 orders, of magnitude greater than that of the smooth fine-grain line from the phosphorus-rich end member.

With gallium arsenide as the starting material, the phosphorus diffusion-conversion mechanism may be postulated as taking place into the surface by a series of discrete steps. Each step would comprise a thin zone, and within that zone it's composition would be uniform. At the state which M6108 BE was prepared, at least five such zones can be identified. Each zone represents an equilibrium condition, since it has a crystalline structure in keeping with it's composition. Since these zones are thin, their diffraction patterns are fine-grained. The central "kernel" of each powder particle of gallium arsenide may have a diameter many times thicker than the thickness of each separate zone. Confirmation of the presence of such striae was obtained photographically. Figure 2 illustrates such striae in specimen M6108 AA, which was phosphorus diffused at 800°C, under 15 atmospheres pressure, for 7 1/2 hours. In the cross section view, the white area is the polished gallium arsenide surface. The gallium phosphide layer lies between 3.1 and 3.2 scale divisions. The lower irregular line at 3.2 represents the innermost boundary of gallium phosphide, as defined by electro-etch staining (which will be described later). The striae are visible, and were very well defined under direct microscopy.

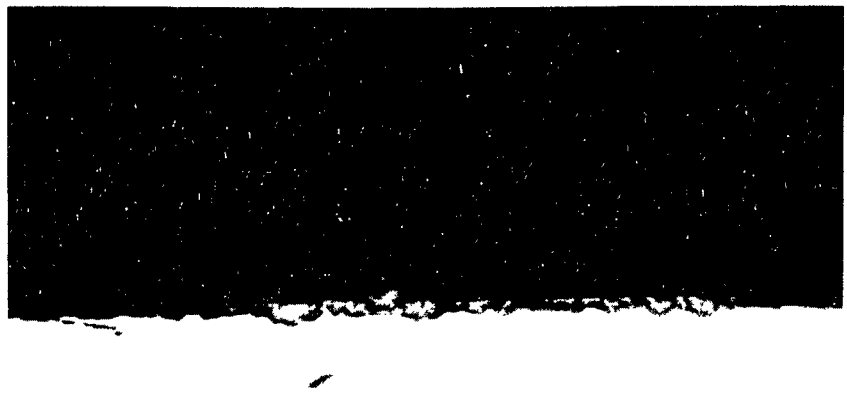


Figure 2. Cross Section View of Variable Gap Wafers Showing Striae.

Note was made that in the heavily diffused specimens of the series, M6108 BG and M6108 BH, the presence of intermediate phases were not found. The implication is that conversion occurred all the way to the center of the 5-micron radius powder particles. This is in agreement with microscopic evidence of wafers diffused simultaneously with the powder specimens in which a layer of gallium phosphide 10 to 15 microns thick was observed and photographed.

Figure 3 illustrates wafer specimen M6108 BH, in cross-section view after etching heavily to define the gallium phosphide layer.

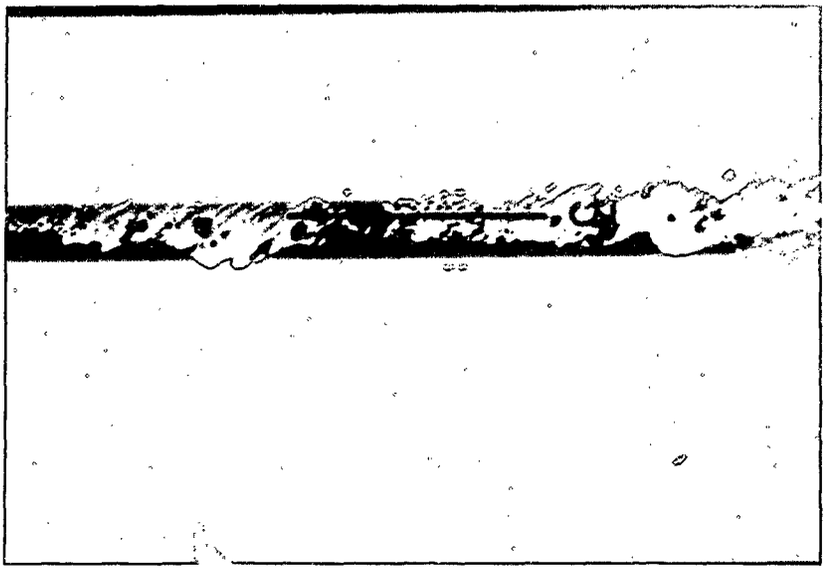


Figure 3. Cross Section View of M6108 BH Wafer Showing 10-Micron Gallium Phosphide Layer. (500X).

Some effort was made to define the diffusion-conversion rate. Table II presents measurements made on variable gap structures of different diffusion schedules.

TABLE 11

Measured Depths of Gallium Phosphide Layers

Identity Numbers	Phosphorus Temp. °C.	Diffusion Pressure-Atm.	Time (Hours)	Depth (Microns)	Percent Concentration By X-Ray
M6108 BE	800°C	15 Atmospheres	2 Hours	----------	50.8
M6108 AA	800°C	15 Atmospheres	7 1/2 Hrs.	About 2	73.0
M6108 BF	800°C	15 Atmospheres	20 Hours	2	81.4
M6108 BG	800°C	15 Atmospheres	50 Hours	2 or 3	85.0
M6108 BH	900°C	20 Atmospheres	50 Hours	10	88.0
M6111 BE	950°C	20 Atmospheres	170 Hours	30	> 90.0
M6111 BF	950°C	20 Atmospheres	340 Hours	50	> 90.0

The data in this table must be interpreted in the light of the following factors:

1. The concentration of gallium phosphide appears to affect the etch resistance of a layer; M6108 BE corresponds to x-ray data indicating 50 percent concentration; it appears to be attacked by the etchant. Etching did not clearly define a layer, although other evidence strongly indicated such was present.

2. The figures listed for M6111 BE and M6111 BF are for the maximum depth. Each wafer had a highly polished face, and an unpolished face. The polished faces indicated 20 and 40 microns, respectively, in depth. The unpolished faces indicated rather large crystalline protuberances grown out of the face, apparently the result of nucleated epitaxial growth. This side had the deeper layer. It is not known which (arsenic or gallium) lattice face this might represent.

3. The number of specimens involved was not statistically large.

4. The conversion process is known to be both pressure and temperature sensitive.

The data does support the following tentative conclusions:

1. The initial rate of diffusion-conversion at pressures in the order of 15 atmospheres, was very rapid, exceeding the diffusion rate of phosphorus at atmospheric pressures, or less, by several orders of magnitude.

2. The initial rapid rate decays more rapidly than the usual error function associated with diffusion processes.

3. The overall diffusion was very non-linear and did not conform to the Boltzman model.

4. The overall depth and concentration appears most dependent upon temperature, secondarily, upon pressure and time, in the pressure region 15 to 20 atmospheres.

It is interesting to note that gallium phosphide crystals were formed at one end of the ampoules during the longer time runs. The ampoules were prepared with phosphorus at one end, specimens in the center, and the other end was sealed under vacuum. Cooling was done slowly, with the sealed end protruding into a cooled zone to condense the phosphorus and prevent deposits of elemental phosphorus on the wafers. At the opposite end of the ampoules, pure gallium phosphide crystals were observed, larger in size in the longer time run. This may offer a different technique of growing gallium phosphide crystals, if the ampoule contained elemental gallium instead of gallium arsenide wafers.

With the objective of preparing shallow layers of very high gallium phosphide concentration, two experiments were made of phosphorus diffusion at slightly more than 30 atmospheres pressure, 800°C for two hours. Powder specimens were included. X-ray patterns were poorly defined and judged not usable for calculation. Cells were observed to have a glass-like film coating. Zinc diffusion resulted in a very thin (estimated of the order of .2 micron) photogenerating surface. In the second experiment the same film was noted and removed prior to zinc diffusion with concentrated HF acid. Cells fabricated thus were poor, and lacked the usual variable gap characteristics. It is possible that this order of

pressure exceeds the disassociation pressure of gallium arsenide and inhibits the conversion process. An alternative explanation contemplates the rapid formation of an SiO_2 film to produce the same result. In the interest of expediency, it was decided to limit the fabrication to 20 atmospheres pressure.

The many possible depths of gallium phosphide layers, further complexed by concentration variables, required limitation if the investigation was to adhere to it's main objectives. Therefore, it was decided to use two types of variable gap structures, i.e., one with shallow, minimal gallium phosphide layer depth as produced by phosphorus diffusion schedules of 800°C - 15 atmospheres, 2 to 10 hours, and the other of fairly deep gallium phosphide structure, as produced by schedules of the order of 900°C - 20 atmospheres, 2 to 50 hours. These two types were considered to bracket the desired structure for the variable gap cell.

D. Zinc Diffusion.

Zinc diffusion as previously reported[2] was carried out in tube furnaces, in ambient hydrogen atmosphere. The specimen and dopant were placed in a clean quartz boat, which was retained in the tube external to the furnace during outgassing and heat up. Temperature was measured by a quartz enclosed thermocouple inserted in the tube to the center of the hot zone. When the desired stabilized temperature was attained, the boat was moved to the center of the hot zone for the desired length of time. Quick cooling was obtained by removing the entire tube from the furnace, the boat moved back to it's former position, and the entire tube was cooled quickly.

The hydrogen atmosphere was maintained until the tube cooled to room temperature. Argon gas was used for purging.

Initial fabrication and testing of single gap cells indicated diffusion schedules of about 600°C for 5 to 6 minutes produced satisfactory junctions about 1-micron deep. Spectral response curves corroborated this order of depth, indicating fairly broad response in the blue region, peaking in the .8-micron wavelength region. Junction depth measurements on cells with fairly deep gallium phosphide layers (10-micron) repeatedly indicated the junction to be at the bottom of such layers. The indication of a more rapid diffusion rate of zinc in gallium phosphide supports the available information in the literature[3].

Table III indicates typical 4-probe measurements of both single gap and variable gap cells at appropriate points of fabrication. All cells had similar polished surfaces, and similar diffusion techniques.

TABLE III

Typical 4-Probe Data of Single Gap and Variable Gap Cells.

Type Cell	No. of Cells	Initial Surface 4-Probe ohms/square	4-Probe ohms-sq. After Phosphorus Diffusion	4-Probe ohms-sq. After Zinc Diffusion	Zinc Diffusion Schedule
Variable Gap,	4	1000 - 1700	6000	5	650°C- 10 Min.
Single Gap,	3	900 - 1200	----	3.5	" " " "
Variable Gap,	8	900 - 1000	7200	7.5	600°C - 5 Min.
Single Gap,	4	700 - 900	----	7.5	" " " "

The table indicates typical increased surface resistance after phosphorus diffusion and approximately equal resistivities, and therefore equal surface carrier concentrations, of both type cells after zinc diffusion.

The indication of a more rapid diffusion rate of zinc in gallium phosphide layers suggested an experimental comparison. Two wafers of similar gallium arsenide, one having a gallium phosphide layer 10 to 12 microns thick, were diffused simultaneously with zinc at 850°C for 10 minutes. As expected, this produced a p-n junction 10-microns deep in the plain wafer, as determined by direct microscopic measurement and by etch-weight loss methods. An electro-etch stain technique, to be described later, produced clear definition of the junction. Similar measurement of the variable gap cell by both cross-section and angle lapped methods is illustrated in Figure 4, and clearly indicated the junction to be about 20-microns deep. Posititve identity of the junction was proved by micro-manipulator probing along the surfaces under 100x magnification, using the vertical microscopic illuminator for illumination. The zinc penetration thus was 10-microns in the plain wafer, and in the variable gap structure was 10 to 12 microns of gallium phosphide plus about 8-microns of gallium arsenide. The indication thus is that zinc penetrated the 10 to 12 micron gallium phosphide layer as easily as 0 to 2-microns of gallium arsenide.

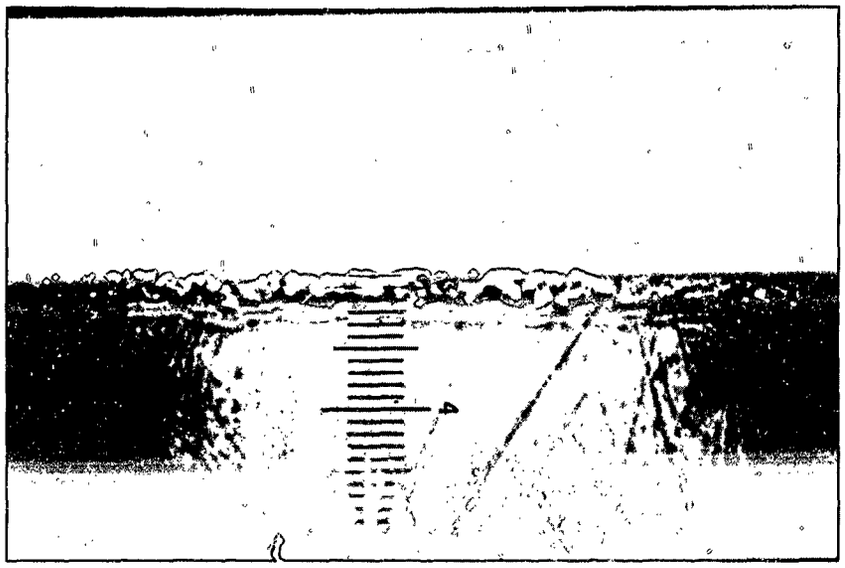


Figure 4. Cross Section View of P-N Junction in Variable Gap Cell. (500x).

The indication, therefore, is at least an order of magnitude faster zinc diffusion in the gallium phosphide layer than in gallium arsenide, perhaps two or three orders. This prompted an effort to reduce the diffusion schedules to produce the junction within the gallium phosphide layers. It was considered advisable to hold the time of diffusion to no less than 10 minutes initially. A series of diffusions were carried out with temperature decreasing from 600°C in 25°C steps. Each group consisted of one lightly phosphorus diffused wafer, one heavily diffused wafer, and one or more plain gallium arsenide wafers. Unfortunately, a materials variable was introduced, as the wafers were from different starting materials. The junction in every case was observed below the gallium phosphide layer. The single gap cells indicate a progressively shallower junction, with Eff_{mp} good at 550 to 500°C.

The phosphorus diffused cells indicate improving Eff_{mp} at 525°C. Further discussion of this study is made under "Evaluation".

A disadvantage of the cool-boat-inserted-into-hot furnace diffusion method is the variable of boat heat up time, estimated to be two to four minutes. Thus a diffusion time of less than six minutes becomes uncertain both as to time and temperature. Similarly, cooling time was difficult to reduce below three minutes. For this reason, a quick heating-quick cooling diffusion apparatus was constructed, similar in many respects to vacuum evaporator apparatus. Tantalum or molybdenum thin-sheet strip heaters were used, and allow independent, rapid heating and cooling of both semiconductor wafers and dopant. Figure 5 illustrates the device. Preliminary trials were promising, heating to 600°C in 60 seconds, and cooling under cold hydrogen gas flow to room temperature in 55 seconds.

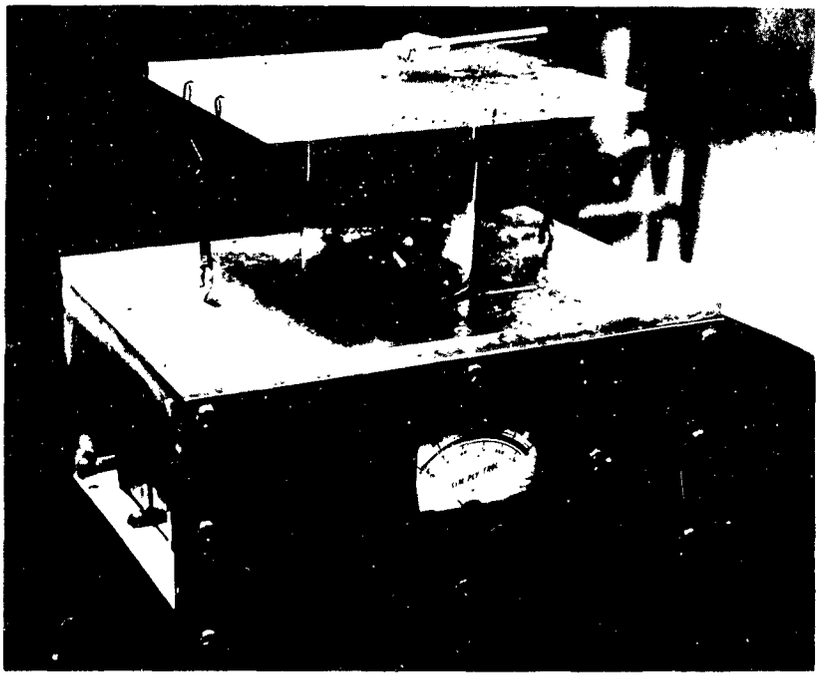


Figure 5. Quick Heating Diffusion Apparatus.

E. Fabrication.

Essential fabrication steps for both single gap and variable gap cells are outlined in Table IV.

TABLE IV

Cell Fabrication Steps and Sequence.

	Variable Gap	Single Gap
(a). Dice material to appropriate size,	X	X
(b). Lap with 600-grit compound to clean saw debris,	X	X
(c). Polish, ending with Linde "B" Compound,	X	X
(d). Clean 10 min. in 100°C concentrated HI acid,	X	X
(e). Thoroughly rinse, ending with de-ionized water, blot dry,	X	X
(f). Seal off in flushed, evacuated ampoule with appropriate charge of high purity phosphorus and diffuse,	X	
(g). Clean in 100°C concentrated HI acid,	X	
(h). Thoroughly rinse, blot dry,	X	
(i). Diffuse zinc to form junction,	X	X
(j). Mask surface with Krylon, lap bottom surface thoroughly with 3-F Compound,	X	X
(k). Plate (electrically) nickel on bottom surface,	X	X
(l). Lap edges with 3-F Compound,	X	X
(m). Dissolve Krylon with 3 baths acetone, ultrasonically, and rinse,	X	X
(n). Clean 2 minutes 100°C HI acid, rinse thoroughly, blot dry,	X	X
(o). Mask all surfaces, sputter platinum collector on top,	X	X
(p). Apply solder to platinum collector,	X	X
(q). Cell ready for evaluation.		

F. Contacting.

Electrically plated nickel contacts were used to contact the n-type bulk gallium arsenide. As deposited, the nickel contact is normally non-ohmic but when heat treated, (inherent in solder coating the nickel) produces good ohmic contact.

Sputtered platinum coated with solder was used to contact the p-type surface. This contact is ohmic, and of low resistance to gallium arsenide. It is ohmic to gallium phosphide, but generally exhibited some resistance in forward current diode characteristics. Variations in this contact resistance may be related to heat applied in coating with solder, hence a brief "alloy-in" heat treatment promises improvment.

G. Evaluation.

1. General.

The evaluation of solar cells included routinely the following procedure:

Photo-parameters, V_{oc}, I_{sc}, Eff_{mp}, were measured either in sunlight, or under 100 milliwatts of light from a Type RFL-2 bulb. In the latter case, incident light was filtered through 1-inch layer of water in a suitable pyrex container and measured with an Eppley pyrheliometer. Short circuit current was measured as a voltage drop across a 1-ohm load resistor.

Diode characteristics were measured using a Techtronix Oscilloscope and curve tracer adapter. When desired, direct current plots were made.

Spectral response data were obtained using a Bausch & Lomb Spectrophotometer of the grating type.

2. Junction Depth and Carrier Concentration.

Junction depth measurements have been greatly facilitated by a technique developed early in this work period. The technique allows non-destructive determination of p-n junction and delineates the gallium phosphide layer simultaneously, using the finished cell. The cell is mounted edge-wise in the groove, sawed in a porcelain block, with one edge protruding slightly. Wire electrodes are brought out the ends of the groove. The protruding edge is lapped and polished. Figure 6 illustrates the mounted, polished cell. A potential is applied across the cell, positive to "p" side, negative to "n" side, sufficient to produce about 5 to 10 milliamperes forward current.

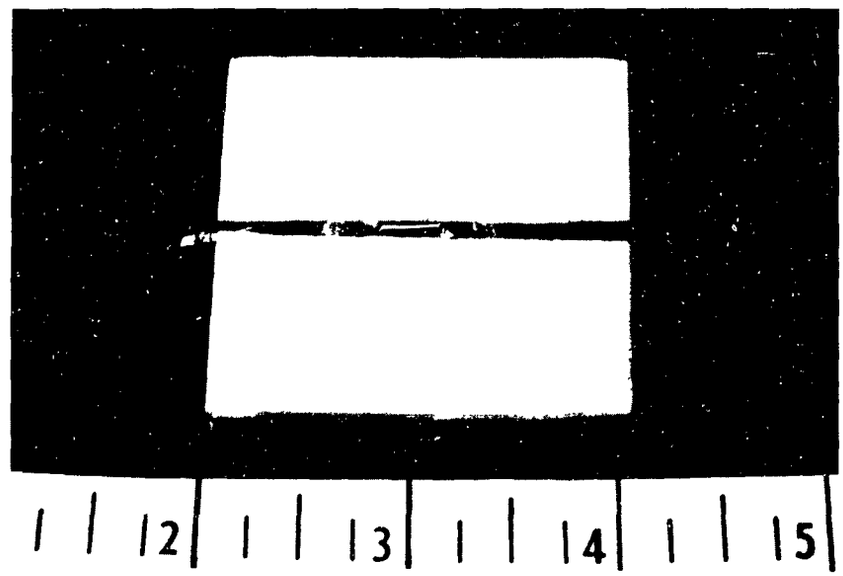


Figure 6. Edge-Mounted Polished Cell Prepared for Electro-Etch-Staining.

A few drops of NH_4Cl of about 4-molar concentration are placed on the exposed edge of the cell for 15 to 30 minutes. The cell is then rinsed carefully and blotted dry. Under magnification, "p" areas are observed stained, "n" areas unstained, with the junction sharply defined by a dark line. Variable gap cells also are found to have the gallium phosphide layers well defined. The same technique is applicable to angle lapped cells. Figure 7 illustrates the junction in a single gap cell, angle lapped, under 500x magnification.

Figure 7. P-N Junction in Angle Lapped Single Gap Cell After Electro-Etch Stain. (500x).

Where no junction is present, the gallium phosphide is also easily and well defined by etching in a solution of 1-$KOH_{(6n)}$ 2-H_2O_2 (30%) 5-H_2O for 15 to 30 seconds. Using either method, direct microscopic measurement to about 1-micron may be made at 500x. Shallower depths require higher magnification and resolution.

Fairly good correlation has been observed between diffusion schedules and the time required to etch single gap cells to zero short circuit current. The solution used is 1-2-5 KOH(6 normal) H_2O_2(30%) H_2O, at room temperature. The general pattern is described in detail in a previous report[4]. Briefly, the I_{sc} shows a rapid increase as the junction is approached, decays quickly to zero as it is dissolved.

Approximate depths may be estimated by time, and as indicated in Table V, the junctions formed at the lower temperature are extremely shallow.

TABLE V

Etch Time to Zero I_{sc} as a Function of Low Temperature Zinc Diffusion for Single Gap Cells.

Diffusion Schedule	Approx. Etch Time to Zero I_{sc}	Est. Junction Depth	Remarks
600°C-10 Minutes,	15 Seconds,	1.0-1.5 Microns	Rise I_{sc} to peak at 6 seconds.
575°C-10 Minutes,	12 Seconds,	.8-1.2 "	Rise to peak I_{sc} at 4 seconds.
550°C-10 Minutes,	8 Seconds,	.6- 1.0 "	Slight rise, drop at 4 seconds.
525°C- 10 Minutes,	6 Seconds,	.5 - .8 "	No rise, drop at 2 seconds.
500°C-10 Minutes,	4 Seconds,	Less than .5 "	No rise; drop 50% in 2 seconds.
475°C-10 Minutes,	2 Seconds,	About .0 Micron	

Carrier concentrations have been determined by 4-probe measurements. A disadvantage has been observed in cells with shallow junctions in that mechanical puncturing of the layers occur frequently, ruining the cell. Consequently, carrier concentration data on the most recent low temperature zinc diffusion work were not obtained. Tentative data indicate that carrier concentration drops rapidly below 500°C diffusion temperatures. Approximately 10^{19} carriers/cm^3 are obtained in either type cell above this temperature.

3. Efficiencies.

Typical photo parameters of single gap, shallow gallium phosphide and deep gallium phosphide variable gap cells are given in Table VI. Data were obtained under 100 milliwatt light input through a 1-inch H_2O filter.

TABLE VI

Typical Photo Parameters of Single Gap, Shallow Gallium Phosphide and Deep Gallium Phosphide Variable Gap Cells.

Type & No. of Cell	Phosphorus History	Zinc History	V_{oc}	I_{sc}/cm^2	Eff_{mp}	V_L
380 S.G.	- - - - - - - - - -	600°C -5 Minutes	.74	14. Ma	6.8%	.60v
382 S.G.	- - - - - - - - - -	600°C -4 "	.72	17. "	7.5%	.54v
402 S.G.	- - - - - - - - - -	540°C- 10 "	.70	14. "	6.15%	.50v
384 V.G.	800°C-15 Atm-3 Hrs.	600°C- 4 "	.65	19. "	5.5%	.41v
396 V.G.	800°C-15 " -2 "	550°C- 10 "	.72	16.5 "	6.3%	.47v
405 V.G.	800°C-15 "	525°C- 10 "	.68	17.4 "	6.1%	.48v
401 V.G.	900°C-20 " -50 "	550°C- 10 "	.42	3.3 "	1.0%	.35v
402 V.G.	900°C-20 " -50 "	550°C- 10 "	.65	2.5 "	1.0%	.44v

It is significant to state that two (#384 and #396) of the variable gap cells were large area cells, enhancing accuracy of measurements, and were of the same order of efficiency as the single gap cells.

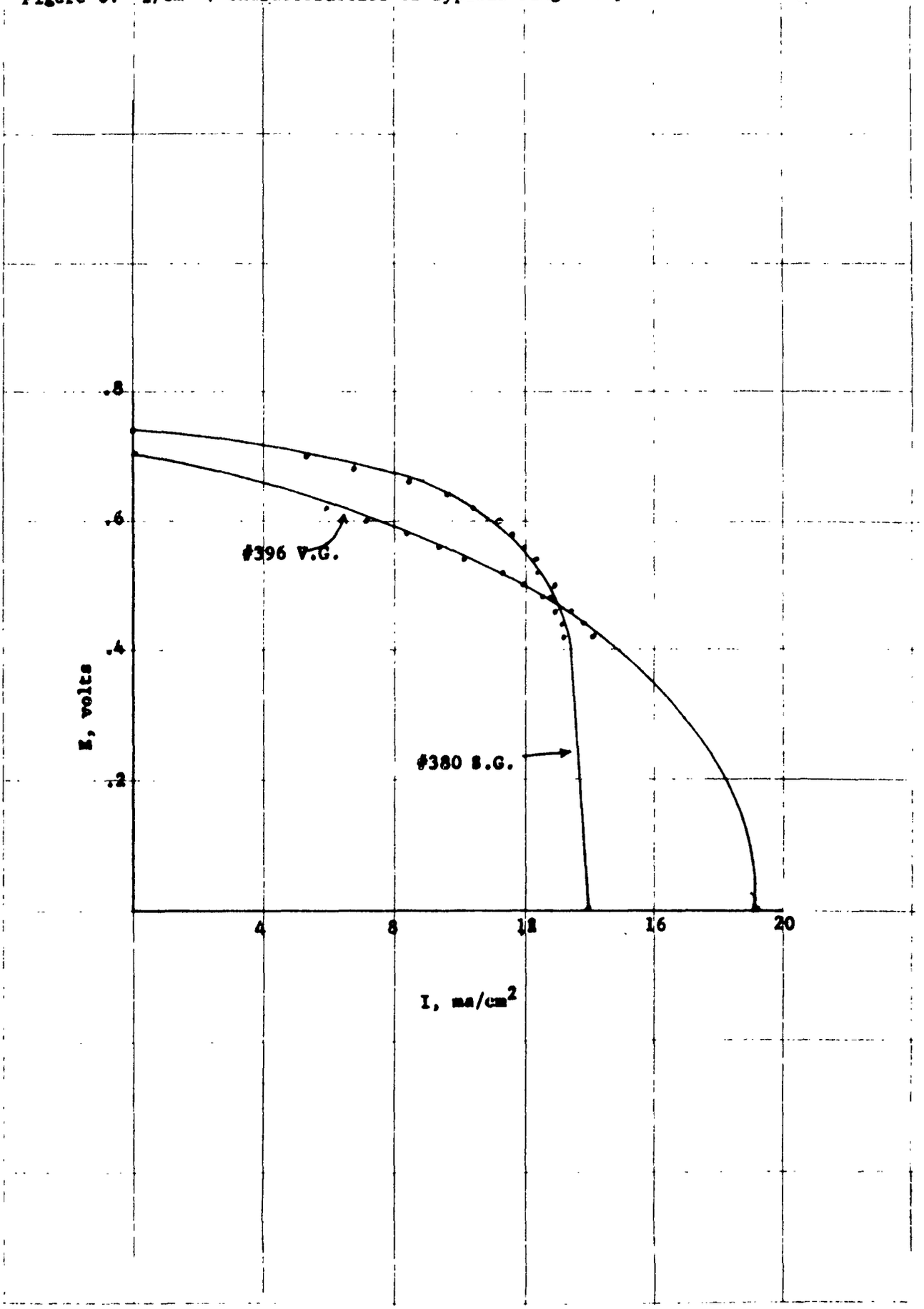
The low efficiencies of Cells #401 and #402, having thick (10 microns) gallium phosphide layers require comment, although full evaluation of this type of cell was not completed. Possible causes include:

(a). Optical absorption in the admittedly thick layer of gallium phosphide.
(b). Poor contacting to gallium phosphide surface.
(c). Partial compensation of the n-type gallium arsenide starting material, due to the long diffusion time. Type change of gallium arsenide bulk material from "n" to "p" type has been observed in wafers diffused similarly for 340 hours; hence, partial compensation could occur in 50 hours. However, the fairly good values of V_{oc} obtained in Cell #402 and others imply little compensation difficulties, especially with reduced diffusion schedules.

The I-V characteristic can be informative in the realm of conversion efficiencies. Typical I-V plots for single gap and variable gap cells are given in Figure 8. Current is plotted per unit area, allowing valid comparison.

The single gap cell characteristics require little comment. The plot is rectangular, and indicates little resistive effects. The plot of the variable gap cell does indicate some series resistance in the slope of the high current region. This is considered to be a composite effect of sheet resistance, contact resistance, and bulk resistance in the gallium phosphide layer, produced by the presence of interstitial phosphorus. Reference is made to subsequent discussion under dark diode data to the resistive effects in this same cell. A contributing factor may well be the high resistivity (.05 ohm-cm) starting material. The fairly good efficiency (6 percent) of this cell appears encouraging to further improvement.

Figure 8. I/cm^2-V Characteristics of Typical Single Gap and Variable Gap Cells.



4. Temperature Characteristics.

The technique for measuring V_{oc}, etc., versus temperature was reported and illustrated in detail in a previous report(2). The results of several such tests upon typical cells agree closely with tentative data given in that report, and are summarized as follows:

The drop in V_{oc} with increasing temperature gave a straight line slope of .00234 + volts/°C for both our own single gap cells and cells fabricated elsewhere.

The drop in V_{oc} with increasing temperature of variable gap cells of the lightly diffused species was regularly less, .00210 v/°C average. Figure 9 is a typical plot of this factor. The I_{sc} versus temperature curves for variable gap cells usually show a more inclined rise than for single gap cells. It is considered this may be related to residual, interstitial, elemental phosphorus in the lattice, which is annealed out during the heating process in testing. Tentative heat treatment of cells in a hydrogen atmosphere appears to increase slightly initial I_{sc} and flatten the rise of I_{sc}. Further evaluation of this point should be made.

The drop in Eff_{mp} versus temperature of the lightly diffused species of variable gap cells is measurably less, but only by a small factor.

Figure 9. V_{oc} versus Temperature of Variable Gap Cell #396 and Single Gap Cell #322

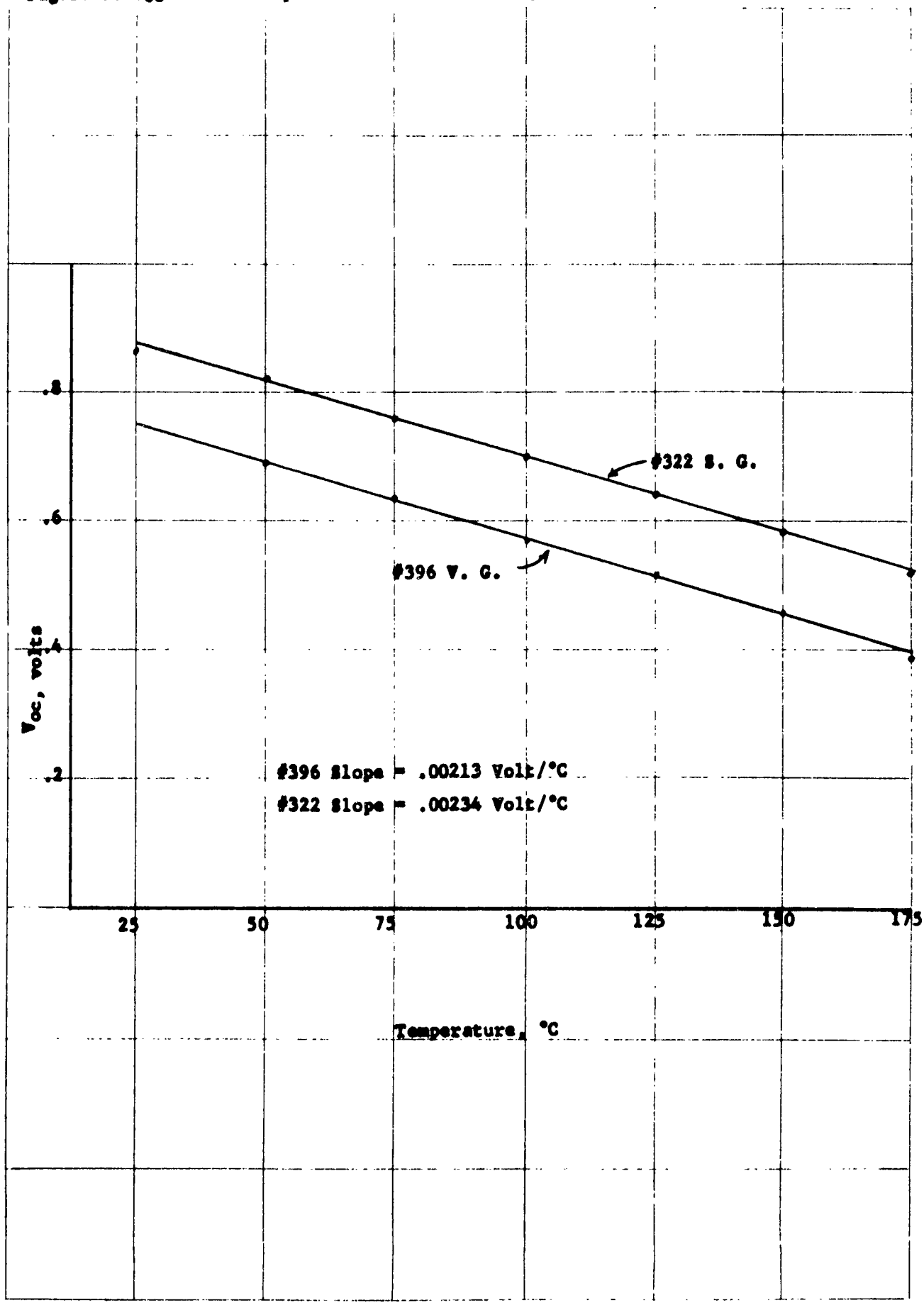
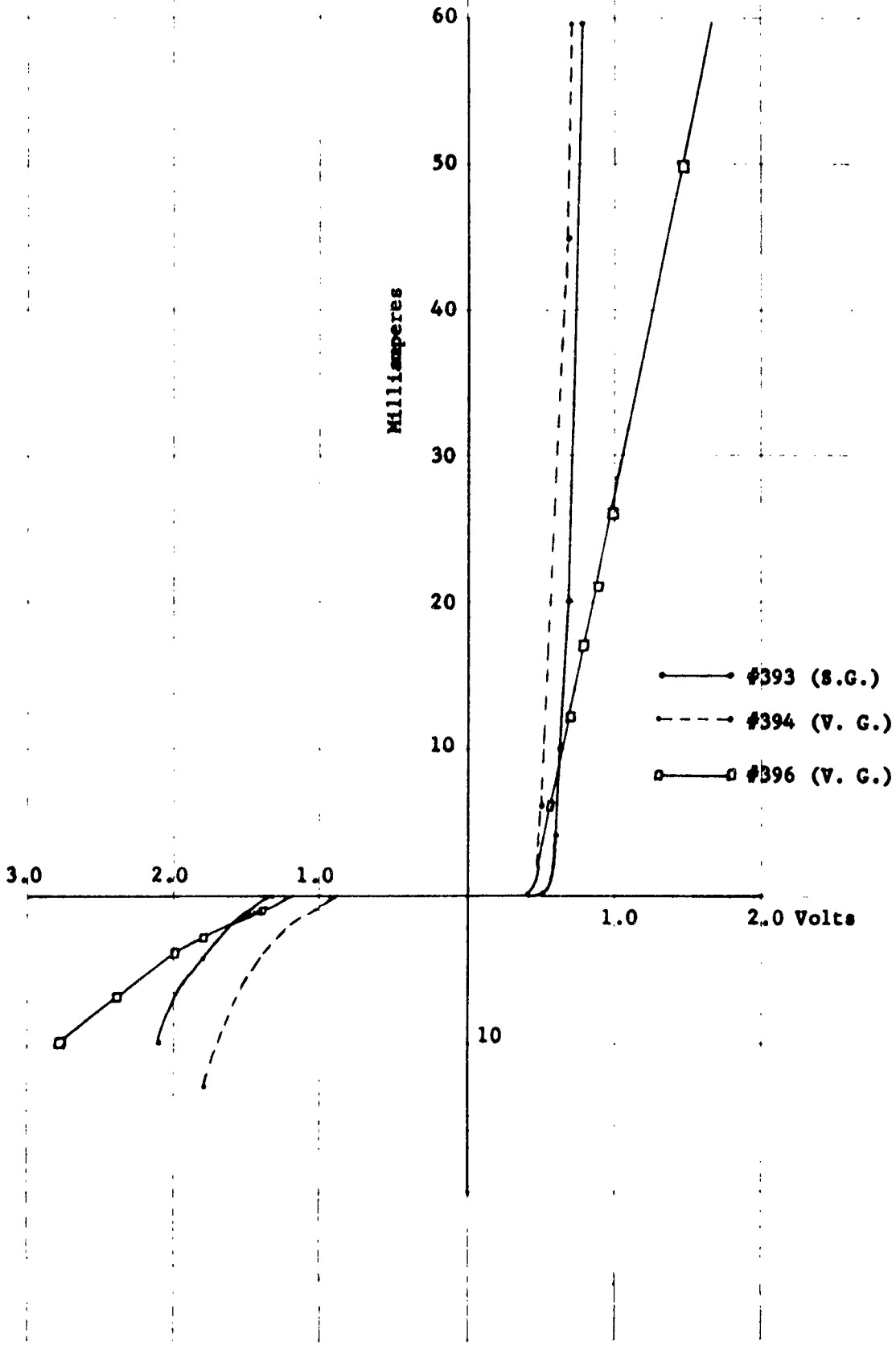


Figure 10. Typical Dark Diode Curves of Single Gap and Variable Gap Cells.



5. Diode Characteristics.

Dark diode data is useful in assessing junction quality and resistive effects. Commonly, both variable gap and single gap cells exhibit good steep forward current slopes, indicating in excess of 60 milliamperes forward current at one volt. Some variable gap cells, although of good efficiency, reflect resistive effects in the forward current slope. Cell #396, variable gap, Eff_{mp} = 6 percent, is such a cell. Figure 10 illustrates it's diode characteristics, which indicates significant contact resistance to the p-type gallium phosphide surface. Also shown is Cell #394 (variable gap) having low forward resistance and Cell #393 (single gap) for comparison. These three are typical of all cells.

6. Spectral Response.

Spectral response was regularly monitored in cells. The response of a series of single gap cells with junctions decreasing in depth show the expected increase in short wavelength response. Good correlation was noted between this blue response and diffusion schedules and junction depths. Figure 11 illustrates the progressive increase of response in the 400 to 600 millimicron region of a series of single gap cells.

It is of interest to note that the single gap cells, although having broad response, retained the high response into the 800 to 850 millimicron region. A characteristic of the shallow gallium phosphide variable gap cells was noted in the peak response occurring at about 700 millimicrons, dropping significantly in the 850 millimicron region. A dramatic difference was noted in deep gallium phosphide variable gap cells, with a pronounced peak, or peaks, occurring in the 400 to 600 millimicron region, and a sharply defined peak at 850 millimicrons.

Figure 12 illustrates typical response curves of the two types.

The peak observed in the short wavelength region is closely related to the band edge of gallium phosphide; that at 850 millimicrons to the band edge of gallium arsenide. This response was easily reproducible in the deep gallium phosphide cells, varying somewhat in amplitude. It is considered significant as a method of monitoring the contribution of current from the higher band gap material.

It is important to note that the response at 850 millimicrons supports strongly the theory of the high band gap "window" principle. This seems reasonable since the gallium phosphide layer is known to be in excess of 10 microns thick, the surface concentration is known to be predominantly gallium phosphide, and the junction is known to be at the bottom of the gallium phosphide layer. The markedly sharp peak at 850 millimicrons supports the conclusion that the "window" transmitted this order of wavelength without great attenuation or absorption.

Figure 11. Spectral Response as a Function of Zinc Diffusion in Single Gap Cells.

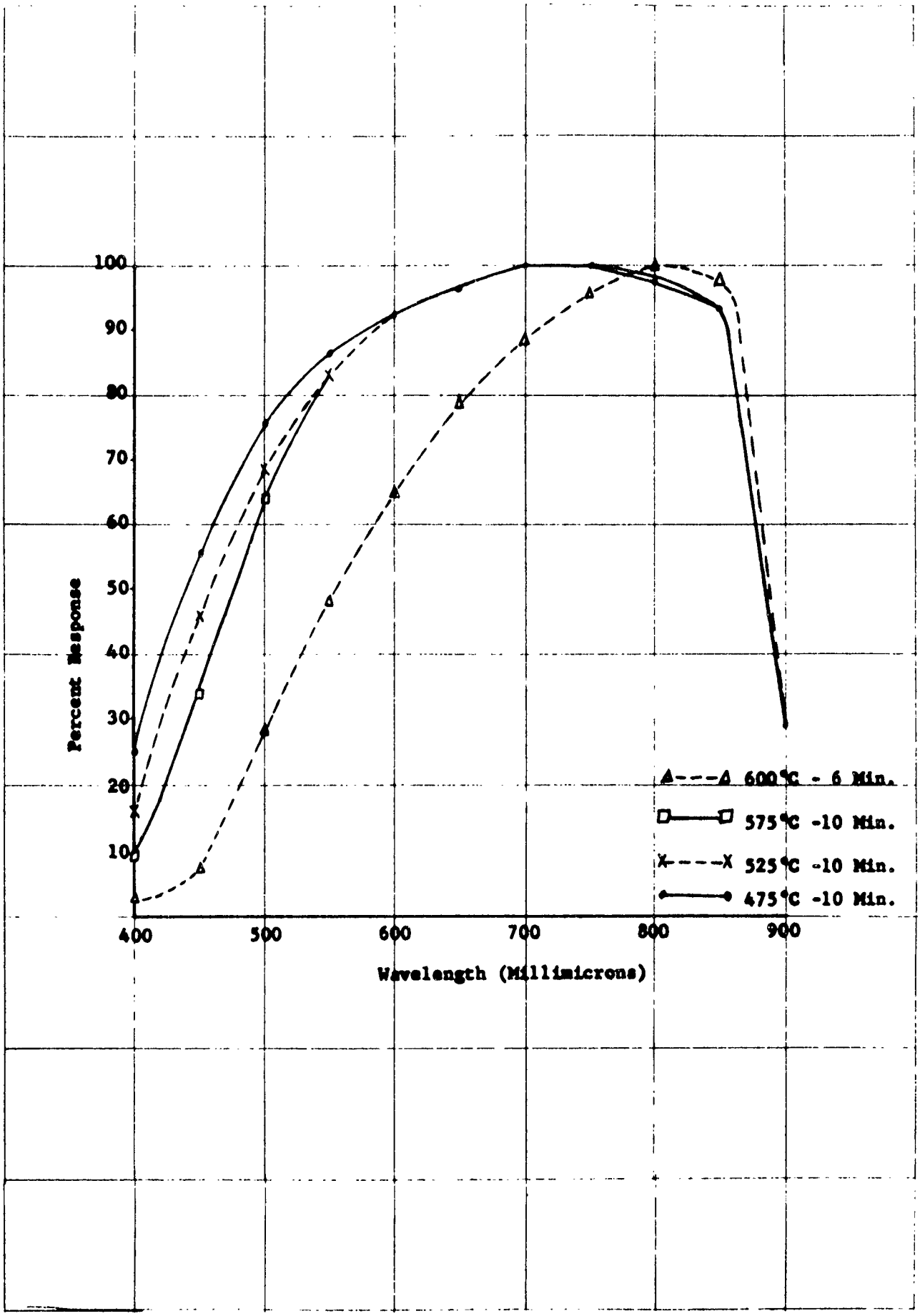
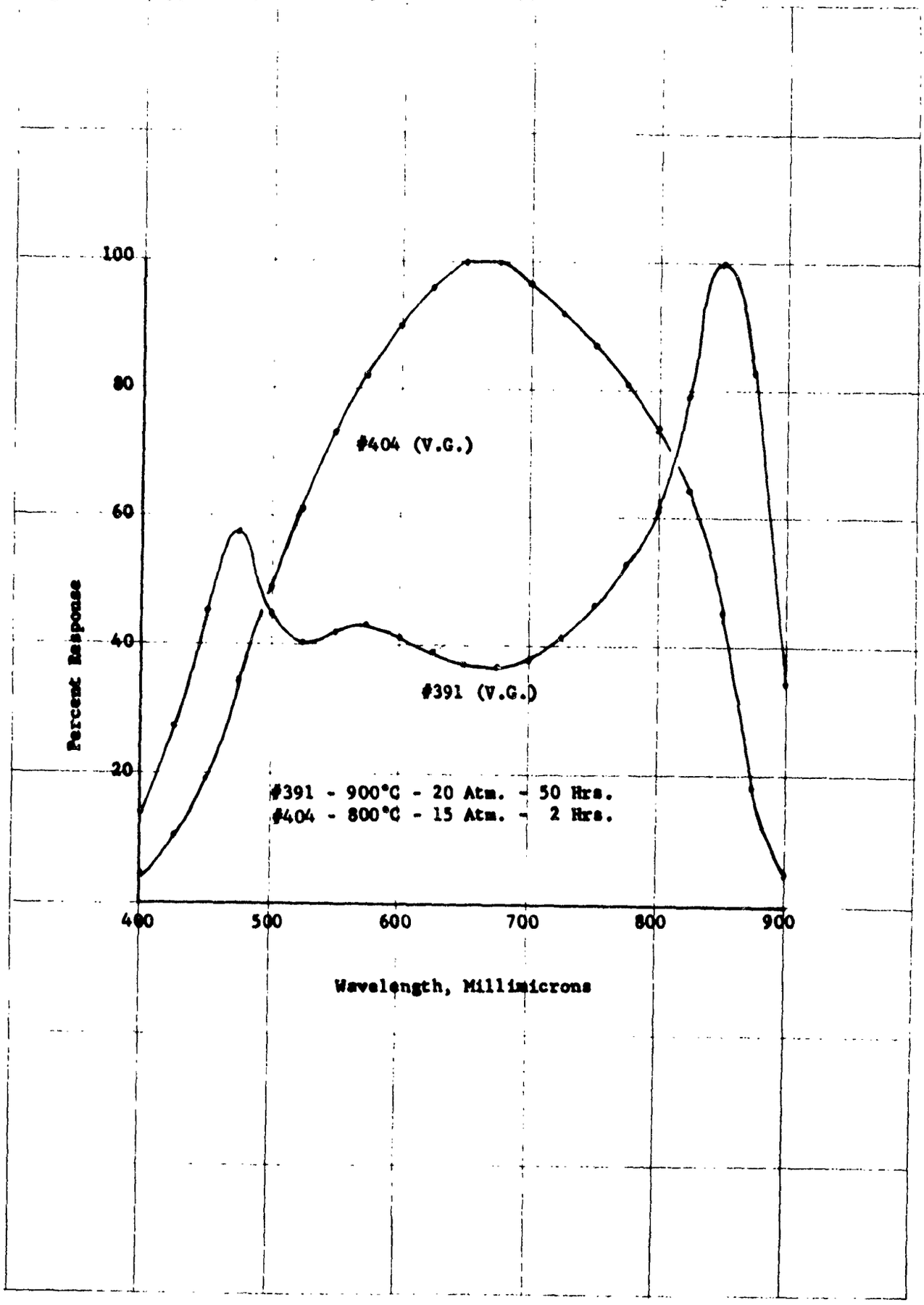


Figure 12. Typical Spectral Response of Two Types of Variable Gap Cells.



V. CONCLUSIONS

It may safely be stated that the compound structure gallium phosphide-gallium arsenide, with intermediate thin transition regions, is produced by solid state diffusion of phosphorus into gallium arsenide at phosphorus pressures in excess of 8 atmospheres.

The diffusion-conversion rate of gallium phosphide formation is pressure and temperature sensitive, is initially more rapid by orders of magnitude than classic phosphorus diffusion, and is extremely non-linear. Significant depth differences between the polished and unpolished side of the same wafer have been observed.

The diffusion rate of zinc into gallium phosphide-gallium arsenide alloys of high gallium phosphide content is significantly faster than in gallium arsenide, and is estimated greater by several orders of magnitude. The "window" effect of the higher band gap layer is valid, and demonstrated by spectral response data included in this report.

Variable gap cells of one type can be of at least equal efficiency as the conventional single gap cell, and it appears feasible to produce good efficiency in deeper gallium phosphide layers.

The technique of electro-etch-staining of variable gap or single gap cells provides very good, non-destructive definition of the junction and gallium phosphide layers.

Variable gap cells of one type exhibits a measurable, although small, advantage in high temperature performance. Further advantage

appears likely with improved techniques, materials and control.

The presence of, and contribution from, the higher band gap component in the variable gap cell is observed in spectral response data.

The contacting of gallium phosphide surfaces for low contact resistance, ohmicity, etc., is possible, although the present system of contacting could be improved.

No well defined relation has been observed between crystal orientation and efficiency as yet. This factor may be obscured by other variables.

VI. FUTURE STUDIES AND RECOMMENDATIONS

It is considered that future studies should include the following:

(a). Pursuit of the deeper gallium phosphide type of cell, with the object of producing a cell combining good efficiency, improved high temperature performance and demonstrative short wavelength response in the 400 to 600 millimicron region related to the band edge of gallium phosphide. It is considered gallium phosphide depth of such a cell might be 2 to 5 microns.

(b). Studies of controls and technique of dopant diffusion aimed at producing the junction in the gallium phosphide layer or below it, rather than the latter only. This may prove especially important in the shallow gallium phosphide type of cell.

(c). The shallow type of cell should be tested, with less emphasis perhaps, pending better control of junction depth.

(d). Investigation, by supplying appropriate specimen cells to Signal Corps Personnel, of radiation resistance and similar characteristics.

(e). Studies intended to evaluate the effect of annealing, under hydrogen, variable gap structures to eliminate any interstitial elemental phosphorus.

(f). Evaluation of the rate of increase of efficiency with increased light intensity. Some evidence has indicated an advantage of the variable gap cells in this report, which would be of importance in solar concentrator applications.

(g). Study and comparison of epitaxially grown gallium phosphide layers, in which no diffusion occurs, and the current solid state diffusion type of variable gap cell.

(h). Study of the "n" on "p" device geometry, using p-type gallium arsenide as starting material.

(i). Studies of a concurrent, cumulative nature which would provide data on -

Materials parameters relative to efficiency.
Crystal orientation with regard to gallium phosphide conversion rates and/or diffusion rates of zinc.

Optimum gallium phosphide concentration and depth for variable gap cells as related to efficiency.

Optimum position of p-n junction relative to efficiency.

VII. REFERENCES

(1). Final Technical Summary Report, Contract DA-36-039-SC-85246. The Eagle-Picher Company.

(2). First Semi-Annual Technical Report, July 10, 1961. Contract Nr. DA-36-039-SC-87408.

(3). Gershenzon, Frosch, Mikulyak = "Precipitation in Gallium Phosphide". Abs. of Electrochemical Society, May, 1961.

(4). Second Technical Summary Report, July 10, 1960, Contract Nr. DA-36-039-SC-85246.

VIII. PERSONNEL

Engineering Time Expended from Feb. 9, 1961 to Feb. 9,1962.

	HOURS
W. E. Medcalf,	272
L. E. Stone,	1,872
J. E. Powderly,	493
George N. Webb,	1,826
Jno. T. Buford,	8
William A. Ames,	82
Kenneth E. Bean,	60
Total Hours,	4,613

AD_____Accession No._____

The Eagle-Picher Company, Chemical & Metals Division, Miami, Okla.
INVESTIGATION OF INTEGRALLY COMPOSED VARIABLE ENERGY GAP PHOTO-VOLTAIC SOLAR ENERGY CONVERTER.
L. E. Stone, G. N. Webb, and K. E. Bean.
Final Technical Summary Report - Feb. 8, 1961 to Feb. 8, 1962.
41 pp - Illus. - Graphs.
Signal Corps Contract Nr. DA-36-039-SC-87408. Unclassified Report.

Gallium phosphide as a discrete phase has been produced in gallium arsenide; concentration of the order of 90 percent identified by x-ray analysis. Photographs of gallium phosphide layers defined by etching are included. Depths of 50 microns were obtained, and diffusion schedules given. A step-type conversion mechanism is postulated; evidence indicates a very non-linear conversion-diffusion rate which is temperature and pressure sensitive. The diffusion rate of zinc in gallium phosphide-gallium arsenide alloys is indicated significantly higher than in gallium arsenide. A non-destructive etch-stain technique using an applied EMF produces excellent definition of p-n junctions and gallium phosphide layers. Efficiency of the same order as single gap cells has been achieved in one specie of variable gap cells. Spectral response data strongly supports the higher band gap "window" effect.

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